

AD-A161 173

STUDY OF FUEL DETERIORATION AND ADDITIVE-INHIBITION(U)
SOUTHWEST RESEARCH INST SAN ANTONIO TX BELVOIR FUELS
AND LUBR. G H LEE ET AL. JUL 85 BFLRF-193

1/1

UNCLASSIFIED

DARK70-85-C-0007

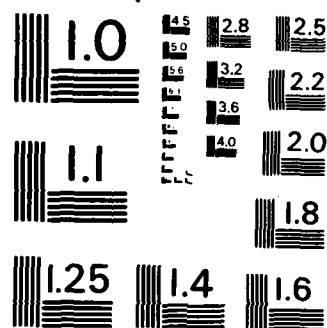
F/G 21/4

ML

END

FORMED

DATE



MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1963-A

AD-A161 173

STUDY OF FUEL DETERIORATION AND ADDITIVE-INHIBITION

INTERIM REPORT
BFLRF No. 193

By

G.H. Lee, II
L.L. Stavinoha

Belvoir Fuels and Lubricants Research Facility (SwRI)
Southwest Research Institute
San Antonio, Texas

Under Contract to

**U.S. Army Belvoir Research
and Development Center**
Materials, Fuels and Lubricants Laboratory
Fort Belvoir, Virginia

Contract No. DAAK70-85-C-0007

Approved for public release; distribution unlimited

July 1985

DTIC FILE COPY

NOV 19 1985

11 18-85 006

Disclaimers

The findings in this report are not to be construed as an official Department of the Army position unless so designated by other authorized documents.

Trade names cited in this report do not constitute an official endorsement or approval of the use of such commercial hardware or software.

DTIC Availability Notice

Qualified requestors may obtain copies of this report from the Defense Technical Information Center, Cameron Station, Alexandria, Virginia 22314.

Disposition Instructions

Destroy this report when no longer needed. Do not return it to the originator.

Unclassified

SECURITY CLASSIFICATION OF THIS PAGE

AD 1161173

REPORT DOCUMENTATION PAGE

1a. REPORT SECURITY CLASSIFICATION Unclassified			1b. RESTRICTIVE MARKINGS None		
2a. SECURITY CLASSIFICATION AUTHORITY N/A			3. DISTRIBUTION/AVAILABILITY OF REPORT Approved for public release; Distribution unlimited		
2b. DECLASSIFICATION/DOWNGRADING SCHEDULE N/A					
4. PERFORMING ORGANIZATION REPORT NUMBER(S) Interim Report BFLRF No. 193			5. MONITORING ORGANIZATION REPORT NUMBER(S)		
6a. NAME OF PERFORMING ORGANIZATION Belvoir Fuels and Lubricants Research Facility (SwRI)		6b. OFFICE SYMBOL (If applicable)		7a. NAME OF MONITORING ORGANIZATION	
6c. ADDRESS (City, State, and ZIP Code) Southwest Research Institute 6220 Culebra Road San Antonio, TX 78284		7b. ADDRESS (City, State, and ZIP Code)			
8a. NAME OF FUNDING/SPONSORING ORGANIZATION U.S. Army Belvoir R&D Center		8b. OFFICE SYMBOL (If applicable) STRBE-VF		9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER DAAK70-82-C-0001; WD 5 DAAK70-85-C-0007; WD 5	
8c. ADDRESS (City, State, and ZIP Code) Fort Belvoir, VA 22060		10. SOURCE OF FUNDING NUMBERS			
		PROGRAM ELEMENT NO. 7T767702		PROJECT NO. AH51FF	TASK NO. (1)
11. TITLE (Include Security Classification) Study of Fuel Deterioration and Additive-Inhibition (U)					
12. PERSONAL AUTHOR(S) Lee, II, George H. and Stavinoha, Leo L.					
13a. TYPE OF REPORT Interim		13b. TIME COVERED FROM Oct 83 TO Sept 84		14. DATE OF REPORT (Year, Month, Day) 1985 July	
15. PAGE COUNT 35					
16. SUPPLEMENTARY NOTATION					
17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)		
FIELD	GROUP	SUB-GROUP	Diesel fuel Mechanism Additive-inhibition		
			Deterioration Fuel Deterioration		
19. ABSTRACT (Continue on reverse if necessary and identify by block number) This interim report summarizes research in support of defining distillate fuel deterioration and additive inhibition. As introductory information, previous work during FY82-83 are summarized. Primary text emphasis is placed on data generated during FY84 involving adherent insolubles, peroxide number, bromine number, and elemental analysis for fuels aged under accelerated conditions. A series of 18 experiments using 3 fuels and 4 additives are reported with primary emphasis on a reference diesel fuel used for engine tests. Observations discussed in the report are summarized as conclusions and recommendations for FY85 ongoing research.					
20. DISTRIBUTION/AVAILABILITY OF ABSTRACT <input checked="" type="checkbox"/> UNCLASSIFIED/UNLIMITED <input type="checkbox"/> SAME AS RPT. <input type="checkbox"/> DTIC USERS			21. ABSTRACT SECURITY CLASSIFICATION Unclassified		
22a. NAME OF RESPONSIBLE INDIVIDUAL Mr. F.W. Schaeckel			22b. TELEPHONE (Include Area Code) (703)664-4594		22c. OFFICE SYMBOL STRBE-VF

DD FORM 1473, 84 MAR

83 APR edition may be used until exhausted.
All other editions are obsolete.

SECURITY CLASSIFICATION OF THIS PAGE

Unclassified

FOREWORD

This report was prepared at the U.S. Army Fuels and Lubricants Research Laboratory (USAFLRL) located at Southwest Research Institute, San Antonio, TX, under Contract Nos. DAAK70-82-C-0001 and DAAK70-85-C-0007, for the period 1 October 1983 through 30 September 1984. Work was funded by the U.S. Army Belvoir Research and Development Center, Ft. Belvoir, VA, with Mr. F. W. Schaekel (STRBE-VF) serving as contracting officer's representative. Project technical monitor was Mr. M. E. LePera, STRBE-VF.

Accession For	
NTIS CRA&I	<input checked="checked" type="checkbox"/>
DTIC TAB	<input type="checkbox"/>
Unannounced	<input type="checkbox"/>
Justification	
By	
Distribution	
Availability Codes	
Dist	Avail and/or Special
A-1	



TABLE OF CONTENTS

<u>Section</u>	<u>Page</u>
I. INTRODUCTION	7
II. EXPERIMENTAL PROCEDURE	10
A. Sample Containers	10
B. Fuel Preparation and Aging	10
C. Additives	11
D. Analysis	11
1. Filterable Particulate Weight	11
2. Particle Size/Number Distribution	11
3. Peroxide Number	12
4. Bromine Number	12
5. Adherent Insolubles	12
6. Elemental Analysis	13
a. Carbon/Hydrogen	13
b. Nitrogen	13
c. Sulfur	13
E. Experimental Matrix	13
III. EXPERIMENTAL RESULTS AND DISCUSSION	13
A. Petroleum JP-5	13
B. Referee Diesel Fuel Containing 1% Sulfur	18
C. Cat 1-H Fuel	21
1. FOA-11 Treated Cat 1-H Fuel	21
2. Neat Cat 1-H Fuel at 95°C	24
3. Neat Cat 1-H Fuel at 80°C	26
4. Neat Cat 1-H Fuel at 65°C	28
5. Cat 1-H With Additive-15 + Additive-B and Single- Package Additive	28
IV. CONCLUSIONS	30
V. RECOMMENDATIONS	33
VI. LIST OF REFERENCES	33

LIST OF ILLUSTRATIONS

<u>Figure</u>		<u>Page</u>
1	Comparison of Calculated and Measured Particulate Weight for Additive-Free JP-5	16
2	Adherent Insolubles Formation as a Function of Time for Additive-Free JP-5	16
3	Peroxide Number as a Function of Time for Additive-Free JP-5	17
4	Schematic of Bottle Placement in Oven	18
5	Comparison of Calculated and Measured Particulate Weight for 1% Sulfur Diesel Fuel	19
6	Peroxide Number Versus Time for 1% Sulfur Diesel Fuel Aged at 95°C	19
7	Bromine Number Versus Time for 1% Sulfur Diesel Fuel Aged at 95°C	20
8	Adherent Insolubles Formation Versus Time for 1% Sulfur Diesel Fuel Aged at 95°C	21
9	Measured Particulate Weight as a Function of Time for Cat 1-H + Additive-11	22
10	Calculated Particulate Weight as a Function of Time for Cat 1-H + Additive-11	22
11	Peroxide Number Versus Time for Cat 1-H + Additive-11	23
12	Adherent Insolubles Formation as a Function of Time for Cat 1-H + Additive-11	23
13	Comparison of Calculated and Measured Particulate Weight for Additive-Free Cat 1-H Aged at 95°C	25
14	Adherent Insolubles Formation Versus Time for Additive-Free Cat 1-H Aged at 95°C	25
15	Comparison of Calculated and Measured Particulate Weight for Additive-Free Cat 1-H Aged at 80°C	26
16	Measured Particulate Weights Versus Time for Additive-Free Cat 1-H Aged at 80°C in Pyrex® Containers	27
17	Calculated Particulate Weight as a Function of Time for Additive-Free Cat 1-H Aged at 80°C in Pyrex® Containers	27
18	Comparison of Adherent Insolubles Formation for Cat 1-H Aged at 80°C With Various Additives Present	28
19	Comparison of Calculated and Measured Particulate Weight for Additive-Free Cat 1-H Aged at 65°C	29
20	Adherent Insoluble Formation Versus Time for Additive-Free Cat 1-H Aged at 65°C	29
21	Comparison of Calculated and Measured Particulate Weight for Additive-Free Cat 1-H at 80°C	31
22	Peroxide Number Versus Time for Additive-Treated Cat 1-H at 80°C	31

LIST OF TABLES

<u>Table</u>		<u>Page</u>
1	FY84 Experimental Matrix	14
2	Elemental Composition of Adherent Insolubles From Neat JP-5	17
3	Average Chemical Composition of Sulfur-Containing Diesel Fuel Derived Materials	20
4	Average Chemical Composition of Cat 1-H Derived Materials	24

I. INTRODUCTION

The stability of middle distillate fuels during storage is an important consideration in both commercial and military fuel supply systems. Deterioration products can cause gum-like deposits in fuel supply systems which can, in turn, lead to filter and injector nozzle plugging.(1)* The requirement for long-term storage stability of military fuels is critical in the prepositioning of fuel supplies where good fuel quality must be maintained.(2)

During the past several years, numerous fuel-related problems have surfaced within U.S. Army facilities.(3) These problems are likely to grow with increased use of heavy crudes, variations in refining techniques, and use of synthetic fuels. Fuel storage stability is also dependent on other factors, such as fuel composition, prior history, additive treatment, container design and/or composition, and environmental conditions.

The reactions taking place within finished fuels and the products formed from these reactions have been studied for many decades. It was pointed out over half a century ago that the presence of di-olefins in cracked distillates were responsible for gum formation.(4) Other works written in the late 1920's and 1930's verify and expand on these findings. A postulation as to the chemical character of gums from heating oils was made approximately a quarter of a century ago.(5) Elemental concentration and infrared and mass spectrometric analyses indicated that these particular sediments were esterified, condensed molecules containing nitrogen heterocycles.

It has been further suggested that sediment-formation proceeds by (1) the oxidation of "reactive hydrocarbons", nitrogen heterocycles, and sulfur heterocycles to their corresponding hydroperoxides, followed by (2) decomposition of these hydroperoxides to aldehydes, then (3) reaction of these aldehydes with other hydroperoxides to form peroxyhemiacetals, which (4) condense, forming insoluble polymers.

* Underscored numbers in parentheses refer to the list of references at the end of this report.

More recent work, in which sediment formation was promoted by adding 2,5-dimethylpyrrole (DMP) to pure hydrocarbons containing no sulfur, as well as to real fuels, has led to the postulation of several structural possibilities for the sediment material.(6)

The sediment-promoting properties of 5-ethyl-2-methylpyridine and 2,5-dimethylpyrrole were examined in separate experiments in which they were added to acid-washed fuel at the 50-ppm level.(7) Sediment formation and peroxide concentrations were increased 20-fold by the addition of the pyridine compound when compared to the pyrrole compound.

Similar results were obtained after producing a stable diesel fuel (Texaco D 454) by removing the polar constituents with silica gel.(8) Fractions were eluted from the silica gel with hexane, toluene, and tetrahydrofuran (THF). After evaporating the solvents, each of the resulting extracts was added to an aliquot of the stabilized fuel. Sediment formation was monitored under storage conditions. Only the THF extract produced substantial sediment formation. Shale-derived jet fuel (Shale I) can be stabilized by acid extractions of the nitrogen bases.(7) Replacing these extracted nitrogen compounds and subsequent aging caused the fuel to form sediments. Also, increased peroxide levels were found in the aged fuel. Mass spectral analysis of the extracted compounds showed the majority to be substituted pyridines and quinolines. Lesser amounts of indoles, pyrroles, and piperidines were also seen.

In other work, soluble sediment precursors from No. 2 home heating oil were characterized by field ionization mass spectrometry.(9) Molecular weight profiles were obtained for residues from fresh and aged fuels which had been concentrated under nitrogen. Increased levels of higher molecular weight components were observed with increased aging. Initially, dimers (~400 MW) are formed, and as aging progresses, the concentration of trimers (~600 MW) was observed to increase.

In addition, it has been demonstrated that certain compounds which do not produce sediment themselves (e.g., isoquinoline) do enhance the activity of other sediment promoters (e.g., dimethylpyrrole).(10)

Recent work at the Naval Research Laboratory (NRL) has produced information on both peroxide formation (11-13) and light scattering (14) in aging DFM spiked with DMP.

The above work and, indeed, virtually all the pertinent literature had addressed the chemistry of particulate and adherent insolubles formation. This was also a goal of the FY82-83 portion of this program. These data were presented at Army Research Office Engine/Fuels Workshops (15,16) and at the Long-Term Storage Stabilities of Liquid Fuels Conference. (17) Interim Report AFLRL No. 168 (18) summarized activity in fuel stability research and included a historical background and bibliography.

The FY84 research effort addressed an area that has been discussed extensively but has generated minimal research activity. That area is concerned with the actual physical growth of insoluble particulate matter and its relationship to adherent insolubles formation.

Of primary interest during FY84 was the study of the kinetics of insoluble particulate formation as a function of temperature and fuel type. This was accomplished through determination of particle size and number relationships as well as filterable weight by ASTM D 2276. A second objective was to examine the potential correlation between gravimetrically determined filterable particulate weight and a mathematical determination using measured particle size and number.

The particle weight per unit volume was calculated from the formula for a sphere, using an assumed particle density. The form of the equation was

$$W = \sum_{n=1}^X N_n \frac{4}{3} \pi r_n^3 d \quad (1)$$

where W is the weight per unit volume of the particles

X is the number of size ranges studied

N_n is the number of particles per unit volume counted in size range n

r_n is the mean radius in size range n

d is the particle density

Adherent insolubles, peroxide number, bromine number, and percent carbon, hydrogen, nitrogen, and sulfur were also measured as a function of aging time and temperature when sufficient material was available. Three fuels and four additives were used in a series of 18 experiments, which are documented in this interim report.

II. EXPERIMENTAL PROCEDURE

A. Sample Containers

Prior to each experiment, all containers were cleaned according to the ASTM D 4057 procedure. This cleaning consisted of a solvent rinse, soap and water wash, water rinse, and oven dry. In 15 of the 18 experiments, 1-liter borosilicate glass bottles were used. In the remaining three experiments, two used a 12-gallon (45-liter) Pyrex® carboy and the third used a 30-gallon (112.5-liter) metal drum.

B. Fuel Preparation and Aging

Three types of fuels were used for these experiments. Cat 1-H was used most often due to its relative instability. A petroleum-based JP-5 specifically purchased with no additives present and a referee diesel fuel containing 1 wt% sulfur were also used. Prior to aging, all fuels were passed through a 0.8- μ m mixed cellulose ester membrane filter. The borosilicate glass bottles were filled with 950 mL of fuel, the carboys were filled with 10 gallons (38 liters) of fuel, and the metal drum was filled with 25 gallons (95 liters) of fuel. Three temperatures were employed in this matrix: 65°, 80°, and 95°C. A sufficient number of borosilicate glass bottles were placed in the temperature-controlled bath to permit periodic removal in pairs throughout the experiment. The 12- and 30-gallon containers were sampled by the removal of 1-liter aliquots. Due to the large volumes of fuel used for the experiment, removal of the aliquots was considered to have an insignificant effect on the overall aging process.

C. Additives

Four additive materials were used in this work. These materials consisted of:

1. Additive-15 containing a rust inhibitor, dispersant, antioxidant, color stabilizer, and metal deactivator. Addition was made to produce a concentration of 82 ppm (25 lb/1000 bbl) in the fuel.
2. Additive-11 containing a dispersant, antioxidant, and metal deactivator. Addition was made at the same concentrations as Additive 15.
3. Single-Package Additive (SPA), a proprietary commercial material which included components similar to those listed in items 1 and 2 above plus a biocide. One mL of additive was placed in one liter of sample.
4. Additive-B, a biocide used in conjunction with Additive-15 to satisfy requirements of MIL-S-53021, was added at approximately 270 ppm.

D. Analysis

1. Filterable Particulate Weight

The quantity of fuel-insoluble particulate matter was determined by ASTM D 2276 procedures modified to use isooctane rather than petroleum ether or refrigerant 113 as the filter rinse solvent.

2. Particle Size/Number Distribution

A HIAC/Royco model PC-320 particle in liquid counter and LAS-346 laser system were used to determine particle size and number distribution in the aged fuels. Six channels were employed with some variation in size range detected by each channel as experience dictated. The final measuring ranges and chan-

nel numbers selected were 0.5-0.8 μm (Channel 1), 0.8-1.0 μm (Channel 2), 1.0-2.0 μm (Channel 3), 2.0-3.0 μm (Channel 4), 3.0-5.0 μm (Channel 5), and $>5 \mu\text{m}$ (Channel 6) apparent particle diameter.

The particle concentration was calculated using the equation:

$$\text{Concentration (mg/L)} = (0.129 N_1 + 0.344 N_2 + 1.590 N_3 + 7.363 N_4 + 30.16 N_5 + 241.274 N_6) \times 10^6 \quad (2)$$

where $N_1 - N_6$ are the number of particles counted in each channel, respectively.

The average of maximum and minimum particle radii detected by each channel was used, and the assumption of 0.9 gm/cm^3 for particle density was incorporated into the calculation of the coefficients in Equation 2.

Five of the channels used were discretely defined. However, the highest channel, Channel 6 ($>5 \mu\text{m}$), is open ended. In these studies, a mean radius of $4.0 \mu\text{m}$ was assumed. However, further work is required to determine the accuracy that this value represents.

3. Peroxide Number

The peroxide number of the fuels was determined using ASTM D 3703.

4. Bromine Number

The bromine number of the fuels was determined using ASTM D 2710.

5. Adherent Insolubles

After removal of the aged fuel from the containers, the interior surfaces were rinsed with heptane to remove residual fuel, followed by a trisolvent rinse composed of equal volumes of toluene, acetone, and methanol (TAM). The TAM rinsings were then placed in a tared beaker and allowed to evaporate to dryness at 150°C in an airjet apparatus (as used in ASTM Method D 381). The

weight of residue was determined in duplicate and reported as adherent insolubles per 100 mL of fuel.

6. Elemental Analysis

a. Carbon/Hydrogen - Total carbon and total hydrogen were analyzed gravimetrically. Samples placed in a horizontal Linberg oven were converted to CO_2 and H_2O which were absorbed on preweighed substrates. The difference in weight is used to calculate percent carbon and percent hydrogen.

b. Nitrogen - Total nitrogen was determined using an Antek model 720/771 pyrochemiluminescence apparatus.

c. Sulfur - Total sulfur was determined using an EDAX X-ray fluorescence spectrometer. Use of this nondestructive technique allows further analyses to be performed on the sample.

E. Experimental Matrix

An overall summary of the experimental work described previously is given in Table 1.

III. EXPERIMENTAL RESULTS AND DISCUSSION

A. Petroleum JP-5

Three experiments were conducted using JP-5 without additives. The first two experiments were conducted less than 1 month apart and were designed to show variation due to aging at 80° and 95°C. Measured and calculated particulate weight, adherent insolubles, peroxide number, and nitrogen content of the gums, were determined for both experiments. Bromine number was determined for the 95°C test, and sulfur concentration of the gums was determined for the 80°C test.

TABLE 1. FY84 EXPERIMENTAL MATRIX

Fuel	Aging Temp, °C	Container	Duration, Days	Determi- nations*	Additives
JP-5 (raw)	95	1-L Pyrex®	36	A-C	None
	95	1-L Pyrex®	21	A-F	None
	80	1-L Pyrex®	42	A-D, F, G	None
High-Sulfur	95	1-L Pyrex®	14	A-G	None
Diesel	95	1-L Pyrex®	14	A-I	Additive-15
Cat 1-H	95	1-L Pyrex®	5	A-D, F, G	Additive-11
Cat 1-H	80	1-L Pyrex®	39	A-D, F, G	Additive-11
Cat 1-H	65	1-L Pyrex®	32	A-D, F, G	Additive-11
Cat 1-H	95	1-L Pyrex®	7	A-D, F	None
Cat 1-H	95	1-L Pyrex®	4	A-C	None
Cat 1-H	80	12-Gal. Pyrex®	23	A-C, F-I	None
Cat 1-H	80	12-Gal. Pyrex® & 30-Gal. Drum	21	A, B	None
Cat 1-H	80	1-L Pyrex®	14	A-C	None
Cat 1-H	80	1-L Pyrex®	23	C	None
Cat 1-H	65	1-L Pyrex®	32	A-D, F, G	None
Cat 1-H	65	1-L Pyrex®	25	A-C	None
Cat 1-H	80	1-L Pyrex®	14	A-D	Additive-15 + Additive-B
Cat 1-H	80	1-L Pyrex®	14	A-D	SPA, Commercial Single-Package Additive

* A Filterable particulate weight
 B Particulate size/number distribution
 C Adherent insolubles weight
 D Peroxide number
 E Bromine number

F Nitrogen content
 G Sulfur content
 H Carbon content
 I Hydrogen content

The third experiment was designed to determine if bottle location within a constant temperature air oven would significantly change the result.

In both the 80° and 95°C experiments, induction periods were noted for both particulate and adherent materials. These periods were 8 and 3 days for particulates and 15 and 4 days for the adherents, respectively, thus indicating particulate formation prior to adherent formation in both cases. The results of these experiments may be seen graphically in Figures 1 and 2. Calculated particulate weight results are consistently lower than the gravimetric data; however, the curve shapes reflect a strong similarity in the data trends.

The behavior of peroxide number (Figure 3) appears to be rather erratic, with the 95°C test eventually exhibiting lower values than the 80°C test. The maximum bromine number was 0.15 at 21 days. Nitrogen and sulfur analyses and the average values for each are shown in Table 2. Reference 12 reports similar tests at 100°C for both petroleum- and shale-derived JP-5. Although the peroxide number results from petroleum JP-5 testing in this laboratory (maximum value 97 ppm) show good agreement with the shale fuel results reported in Reference 12, the petroleum JP-5 results do not show good agreement. The petroleum JP-5 results in Reference 12 indicated a maximum value of 8 ppm.

A schematic diagram of the air bath bottle locations in another experiment with JP-5 is shown in Figure 4. An obvious trend in apparently high adherent insolubles weight follows the middle row and right rear samples (bottles B, E, H, I). Three of these four samples were also the only ones to exhibit filter plugging in the ASTM D 2276 test.

Thermocouple placement did not indicate undue temperature fluctuations at these points; therefore, light through the vent opening may be an explanation for these results. The vent has since been rerouted to prevent external light from entering the heating chamber.

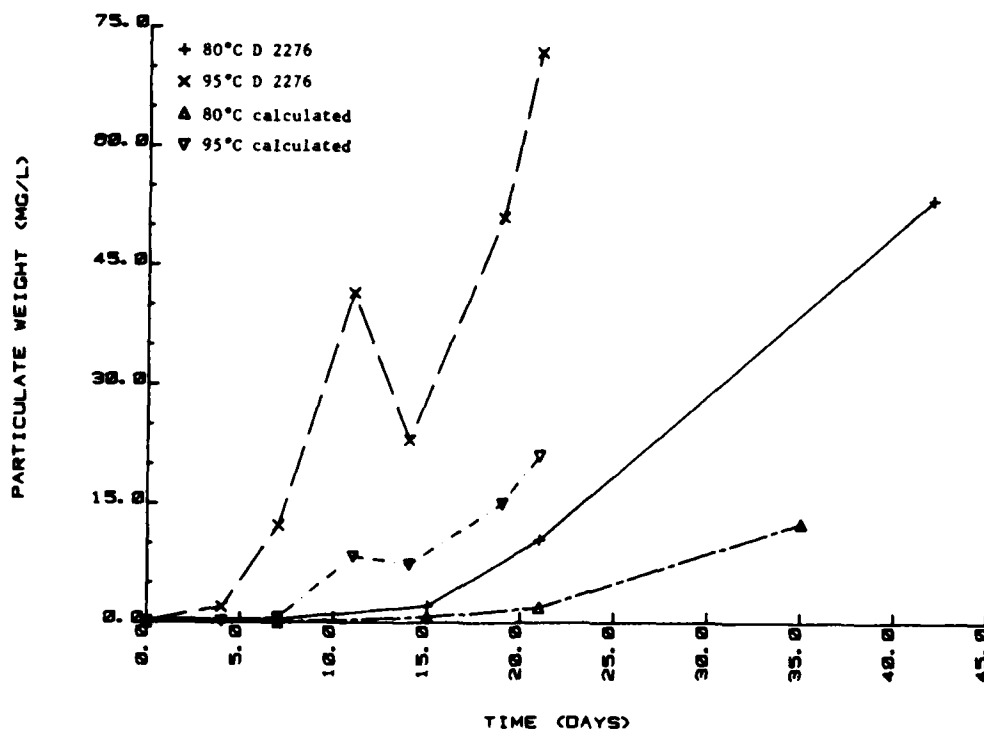


FIGURE 1. COMPARISON OF CALCULATED AND MEASURED PARTICULATE WEIGHT FOR ADDITIVE-FREE JP-5

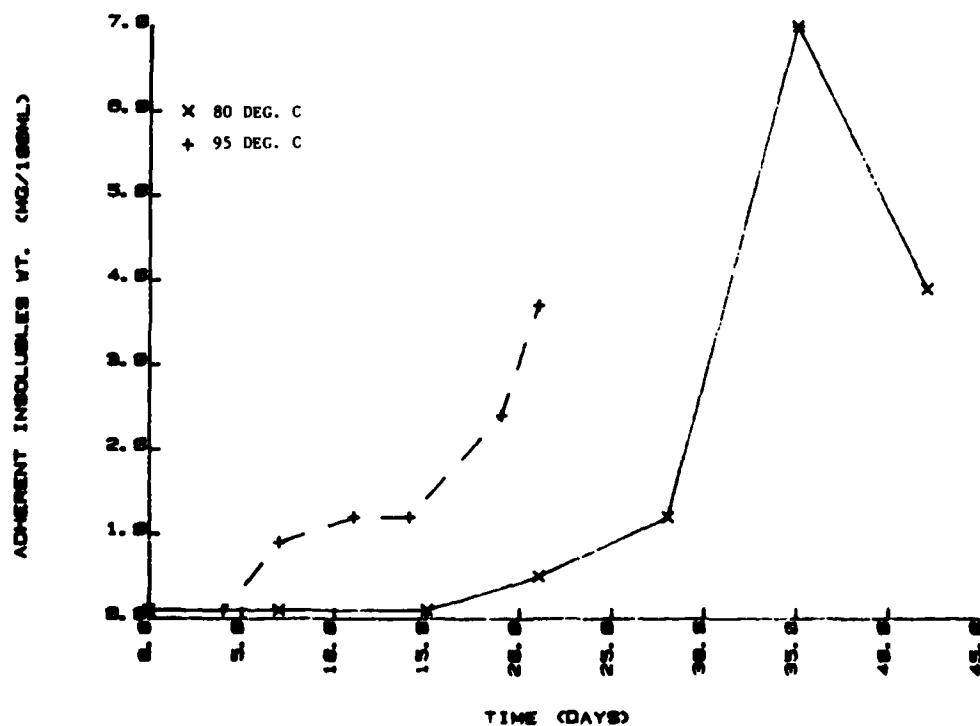


FIGURE 2. ADHERENT INSOLUBLES FORMATION AS A FUNCTION OF TIME FOR ADDITIVE-FREE JP-5

TABLE 2. ELEMENTAL COMPOSITION OF ADHERENT
INSOLUBLES FROM NEAT JP-5

Day	80°C		95°C
	%N	%S	%N
4			2.9
7			2.6
11			2.4
14			2.7
19			2.4
21	3.1		3.1
28	3.7		
35	2.6	2.8	
42	1.9	1.3	
Average	2.8	2.0	2.7

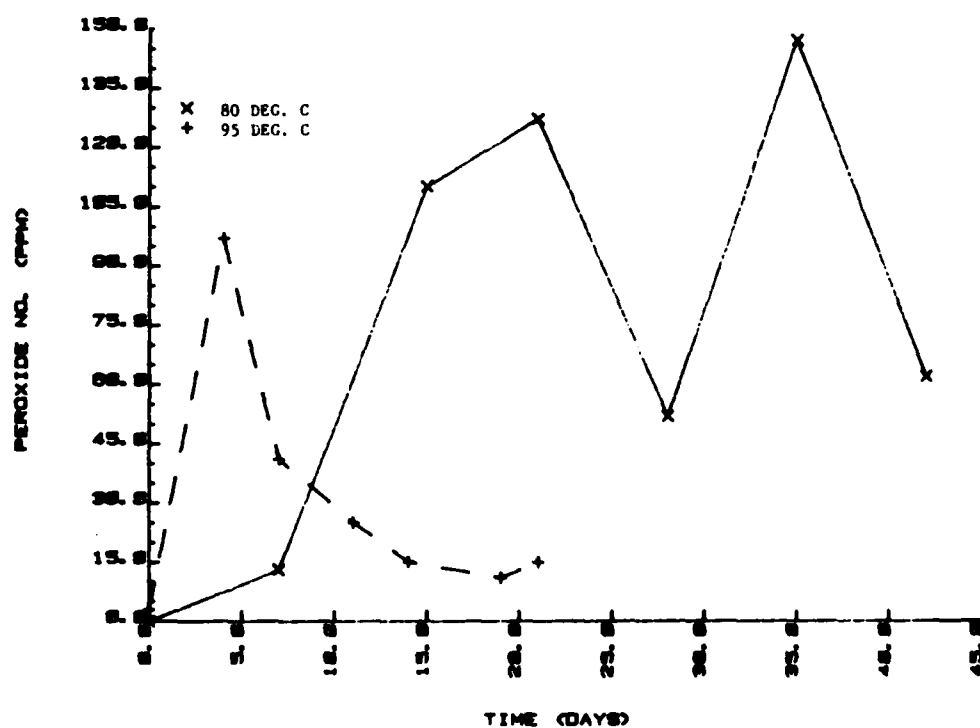


FIGURE 3. PEROXIDE NUMBER AS A FUNCTION OF TIME
FOR ADDITIVE-FREE JP-5

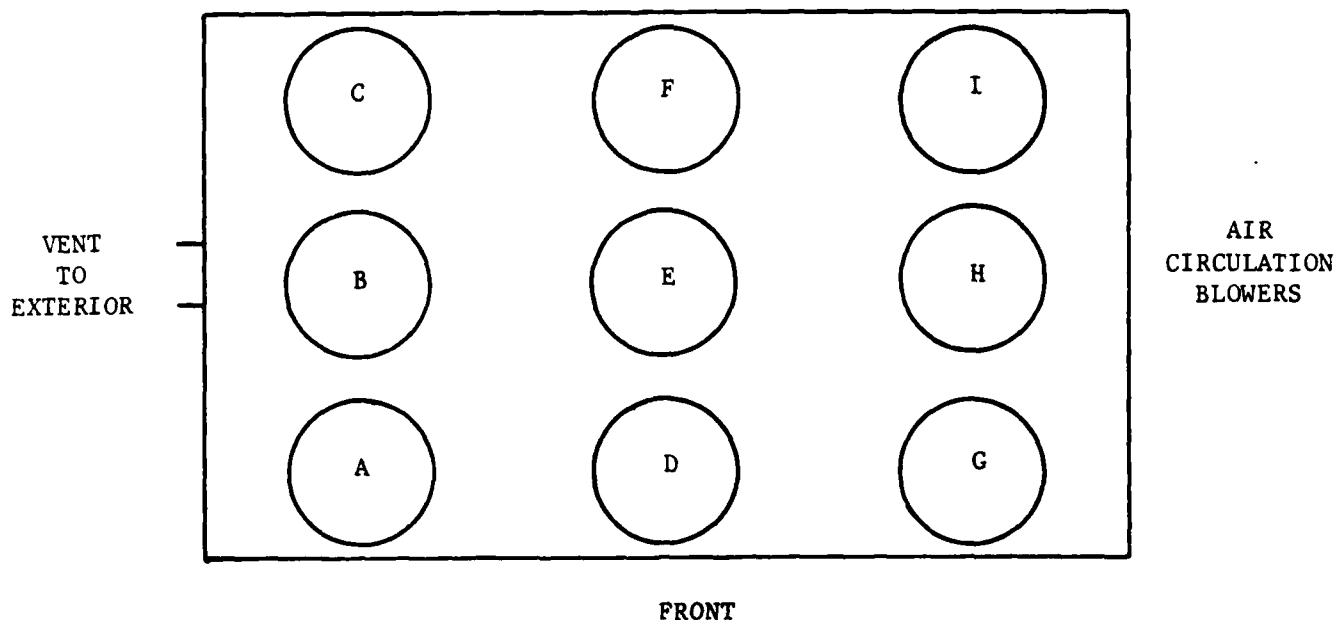


FIGURE 4. SCHEMATIC OF BOTTLE PLACEMENT IN OVEN

B. Referee Diesel Fuel Containing 1% Sulfur

At 95°C, two experiments with and without Additive-15 were conducted. ASTM D 2276 (filterable particulate weight), calculated particulate weight, adherent insolubles weight, peroxide number, bromine number, and N and S concentrations were determined for each test. The C and H content of the adherent materials was also determined for the additive-containing fuel.

The D 2276 values for the additive-treated fuel were generally lower than for the nontreated fuel. The same was true for the calculated particulate weight (Figure 5). However, in both cases the calculated weights were lower than the respective D 2276 values. Peroxide and bromine numbers (Figures 6 and 7) were also lower for the additive-treated fuel. The curves for both experiments parallel each other, including a peak maximum at 4 days in the peroxide number determination. Conversely, adherent insolubles formation was greater for the additive-treated fuel (Table 3 and Figure 8), probably due to the smaller average particle sizes and thus a greater diffusion capability.

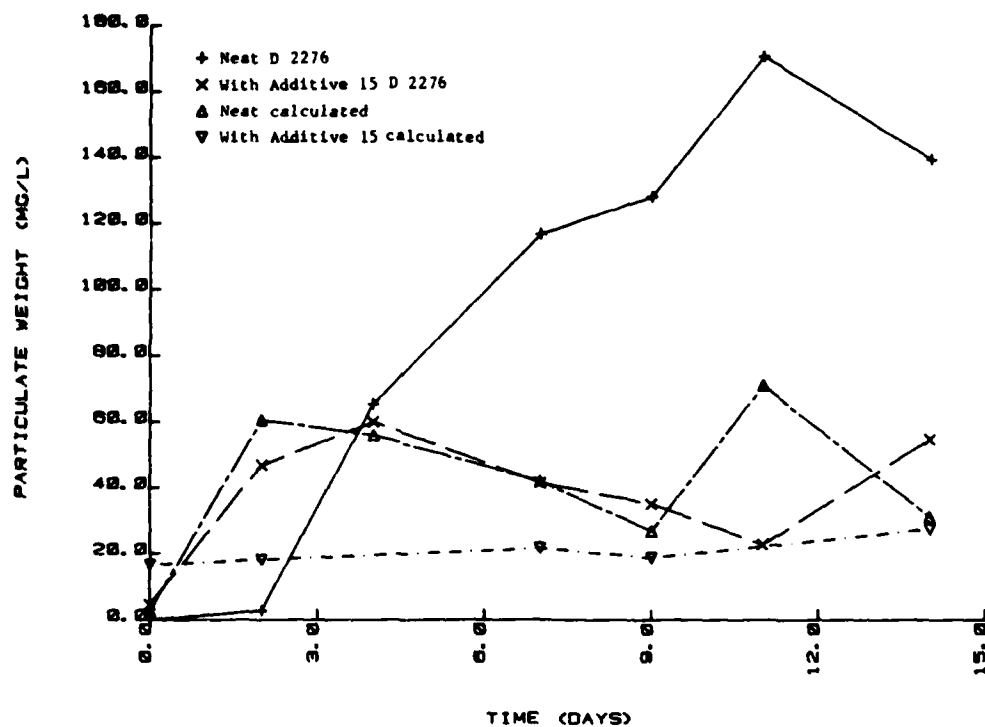


FIGURE 5. COMPARISON OF CALCULATED AND MEASURED PARTICULATE WEIGHT FOR 1% SULFUR DIESEL FUEL

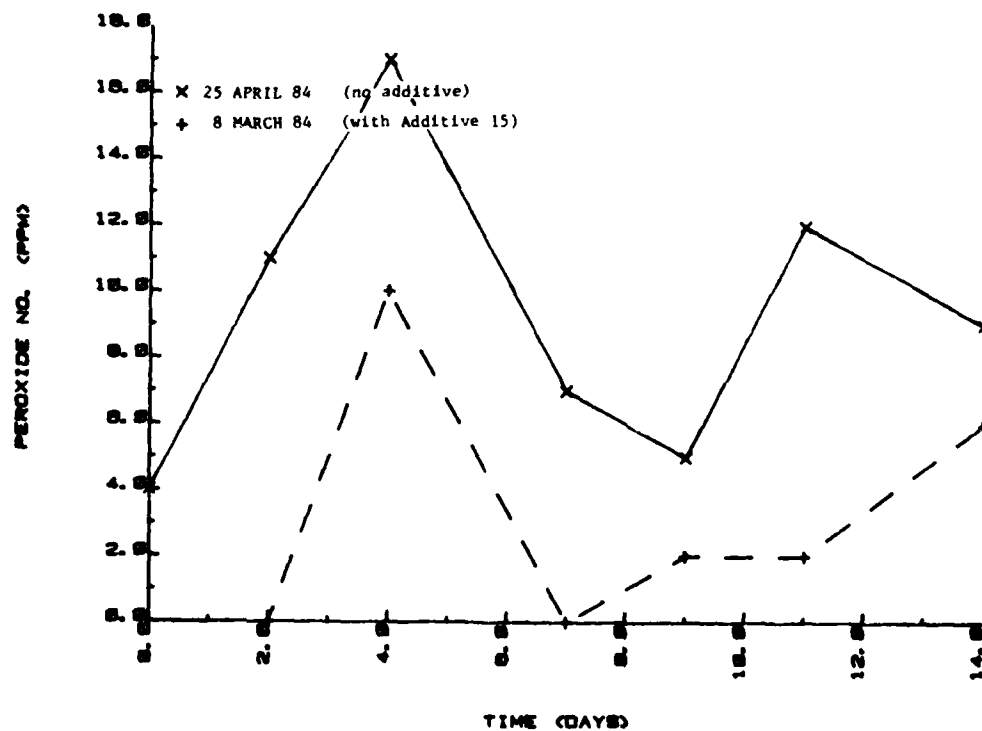


FIGURE 6. PEROXIDE NUMBER VERSUS TIME FOR 1% SULFUR DIESEL FUEL AGED AT 95°C

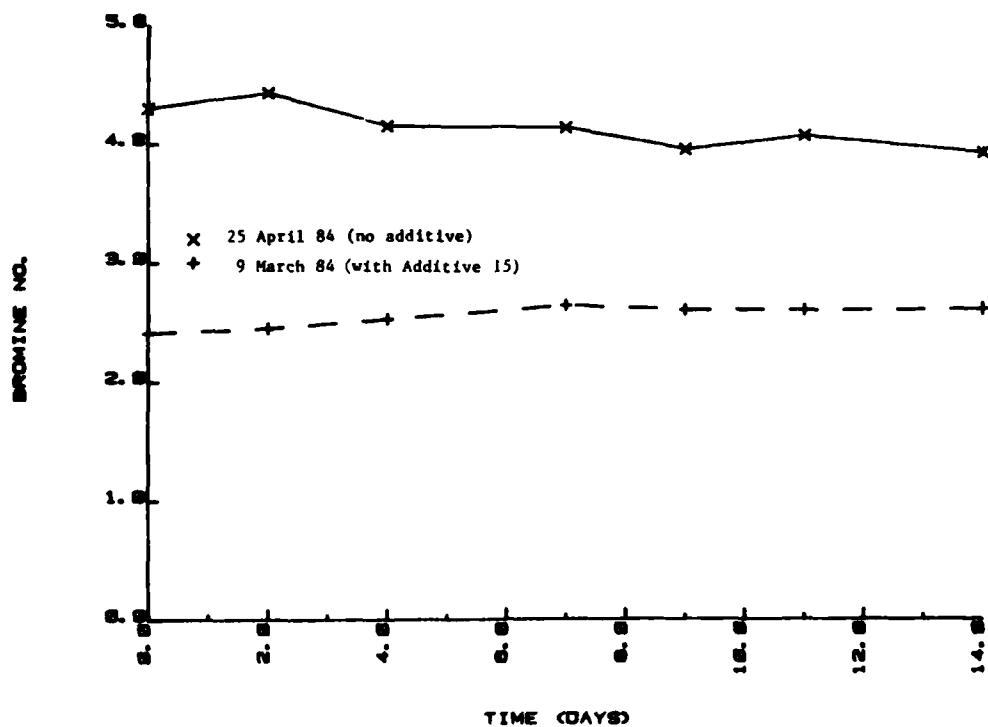


FIGURE 7. BROMINE NUMBER VERSUS TIME FOR
1% SULFUR DIESEL FUEL AGED AT 95°C

TABLE 3. HIGH-SULFUR DIESEL FUEL

Chemical Composition:

<u>Fuel</u>	<u>%C</u>	<u>%H</u>	<u>%S</u>	<u>%N</u>
Neat	--*	--	1.11	--
Additive-treated**	--	--	1.11	0.0075

Adherent Insolubles

Average

Neat	--	--	6.9	3.7
Additive-treated	67.1	6.3	7.6	4.1

* Not determined

** Fuel treated with 25 pounds of Additive-15 to 1000 barrels of fuel

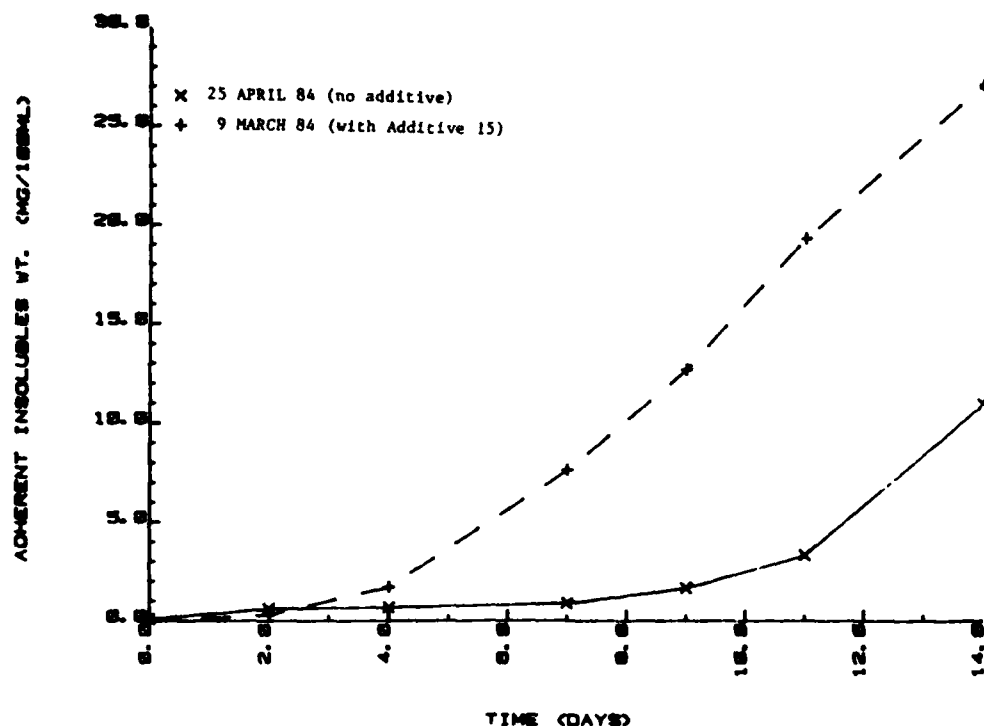


FIGURE 8. ADHERENT INSOLUBLES FORMATION VERSUS TIME FOR 1% SULFUR DIESEL FUEL AGED AT 95°C

C. Cat 1-H Fuel

1. Additive-11-Treated Cat 1-H Fuel

Started less than 6 weeks apart, the three experiments indicate differences between 65°, 80°, and 95°C storage. Analyses include filterable particulate weight by ASTM D 2276, calculated particulate weight, adherent insolubles weight, peroxide number, and N and S concentrations. Except for the elemental concentrations, the measurements made at 95°C exceeded significantly those made at 65°C. The data for 80°C were intermediate in magnitude. Peroxide numbers at 80°C were similar to those of the petroleum JP-5 at 100°C as reported in Reference 12. These data are shown in Figures 9 through 12. Values for the elemental composition of the fuel and adherent insolubles are shown in Table 4. Data for qualitatively measured and calculated particulate correlations are remarkably similar for all three temperatures. As with

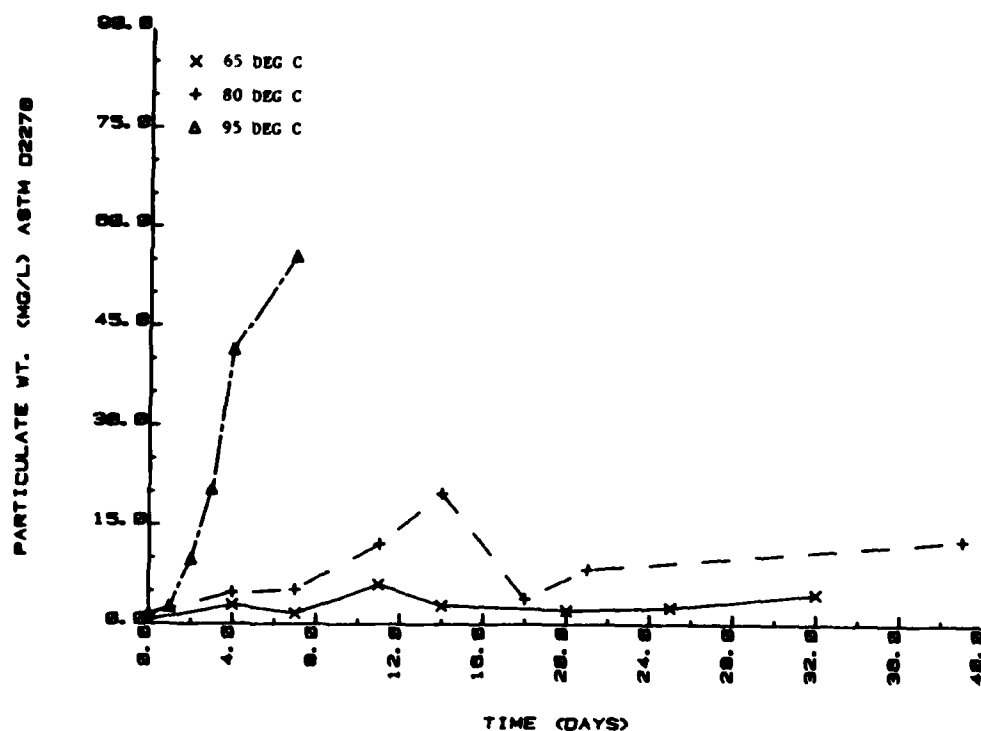


FIGURE 9. MEASURED PARTICULATE WEIGHT AS A FUNCTION OF TIME FOR CAT 1-H + ADDITIVE-11

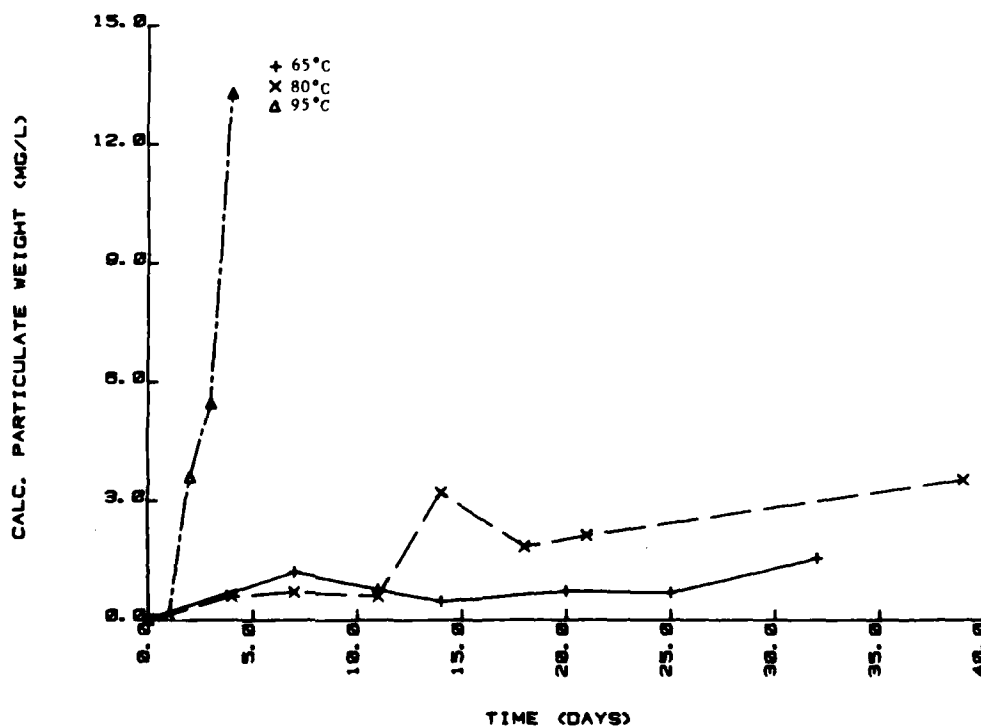


FIGURE 10. CALCULATED PARTICULATE WEIGHT AS A FUNCTION OF TIME FOR CAT 1-H + ADDITIVE-11

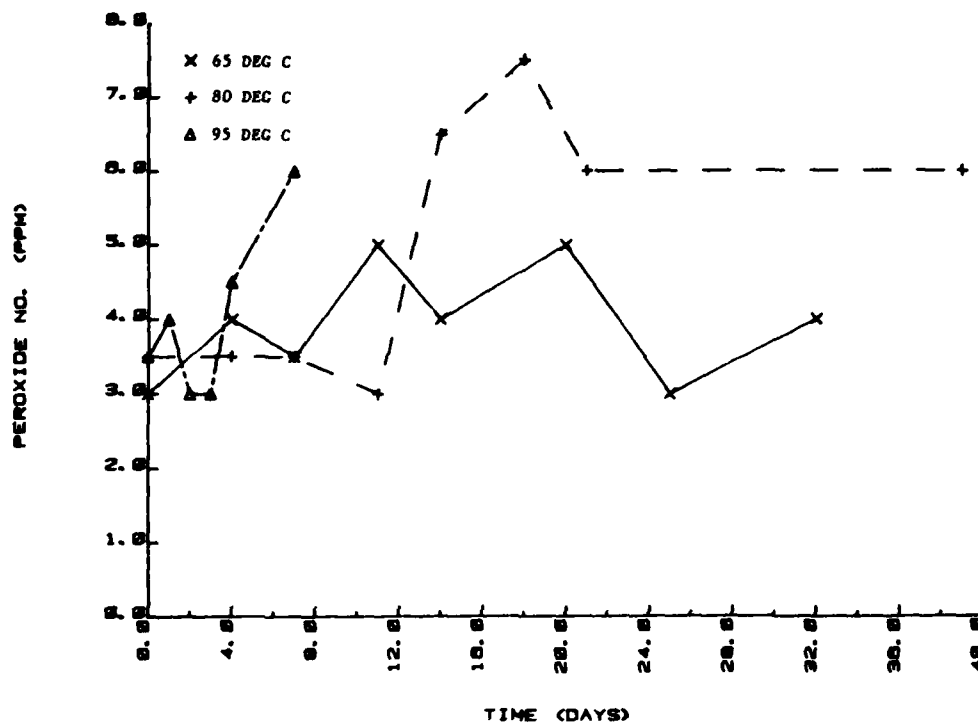


FIGURE 11. PEROXIDE NUMBER VERSUS TIME FOR CAT 1-H + ADDITIVE-11

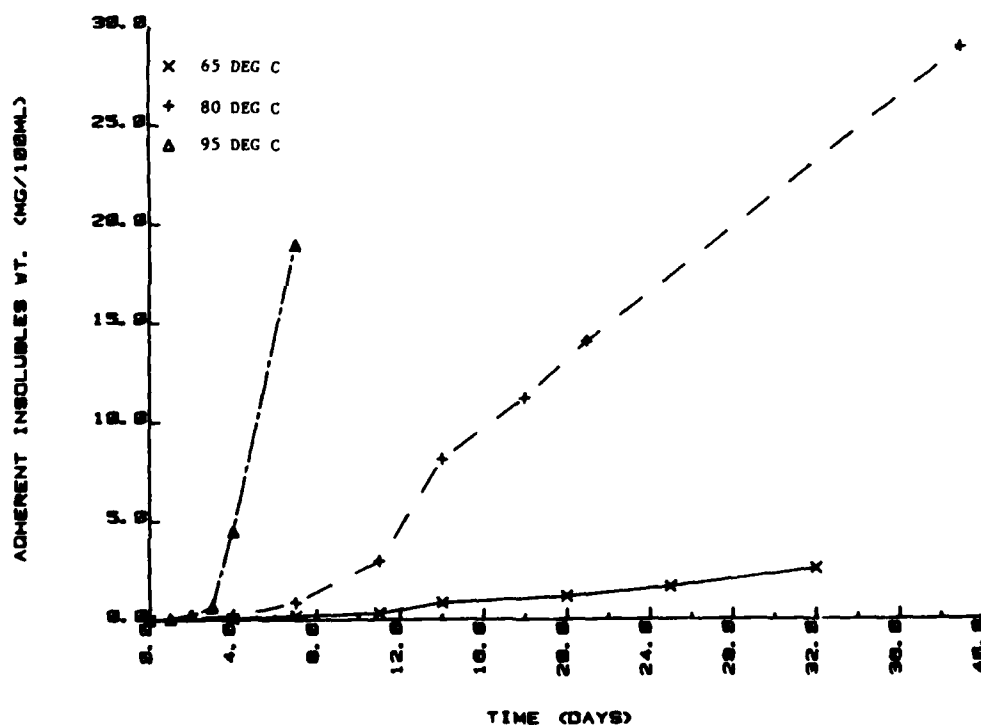


FIGURE 12. ADHERENT INSOLUBLES FORMATION AS A FUNCTION OF TIME FOR CAT 1-H + ADDITIVE-11

TABLE 4. AVERAGE CHEMICAL COMPOSITION OF CAT 1-H DERIVED MATERIALS

Sample	°C	%H	%N	%S
Neat fuel	86.35	13.04	0.005	0.42
Adherent insolubles from neat fuel				
65°C	--*	--	4.4	8.9
80°C**	64.57	6.66	4.5	7.82
95°C	--	--	3.5	8.0
Fuel + Additive-11				
65°C	--	--	3.3	15.5
80°C	--	--	3.1	9.2
95°C	--	--	3.3	9.6
Fuel + Additive-15/ Additive B				
80°C	--	--	3.8	--

All containers were 1-liter Pyrex® except as noted

* Not determined

** 12-gallon Pyrex® carboy

previous measurements, the calculated results are lower than the measured, in this case, by approximately a factor of five.

2. Neat Cat 1-H Fuel at 95°C

Analyses in these two experiments consisted of filterable particulate weight by ASTM D 2276, calculated particulate weight, and adherent insolubles weight. Peroxide number and nitrogen content were also measured for one of the experiments. These tests were run approximately 4 months apart, and they track each other quite well, indicating minimum effect of sample storage on fuel quality. Again, the calculated results are lower than the gravimetric results for particulate concentration, although qualitatively the data are consistent. These data are presented as Figures 13 and 14. Nitrogen and sulfur concentrations are shown in Table 4.

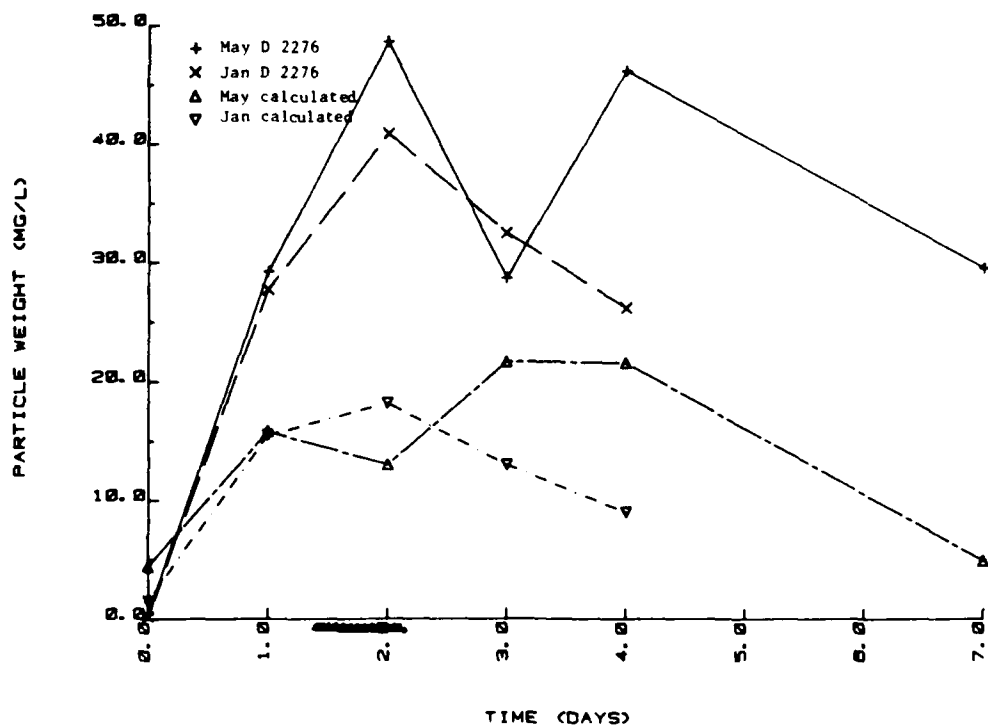


FIGURE 13. COMPARISON OF CALCULATED AND MEASURED PARTICULATE WEIGHT FOR ADDITIVE-FREE CAT 1-H AGED AT 95°C

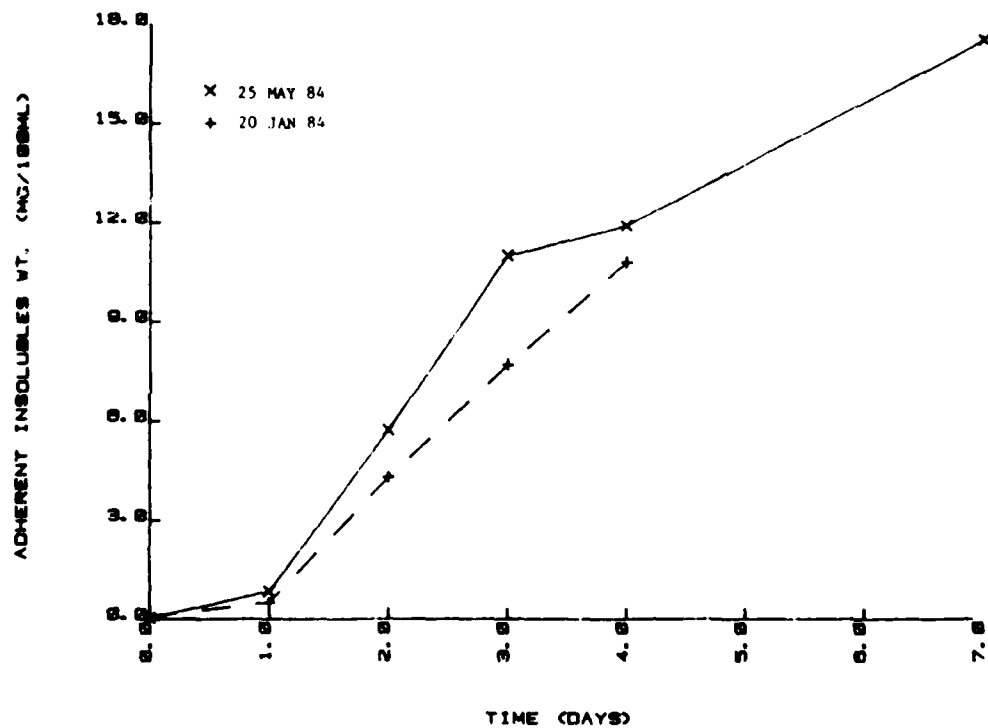


FIGURE 14. ADHERENT INSOLUBLES FORMATION VERSUS TIME FOR ADDITIVE-FREE CAT 1-H AGED AT 95°C

3. Neat Cat 1-H Fuel at 80°C

This category contains the most extensive variety of experimental containers; 1-liter Pyrex® bottles, 12-gallon Pyrex® carboy, and 30-gallon metal drum. Duplicate experiments using the 12-gallon (45-liter) container yielded calculated and gravimetric values that corresponded reasonably well. A comparison of data from simultaneous testing of the 12-gallon (45-liter) Pyrex® container and the 30-gallon (112.5-liter) metal drum also indicated minimal variation in both D 2276 and calculated data. These data are represented in Figures 15 through 17. Figure 18 compares the rate of adherent gum formation for a neat fuel with fuels to which Additive-15, Additive-11, and the SPA have been added. The reduction of adherent insolubles formation in the additive-treated fuels is quite significant. Elemental data may be found in Table 4.

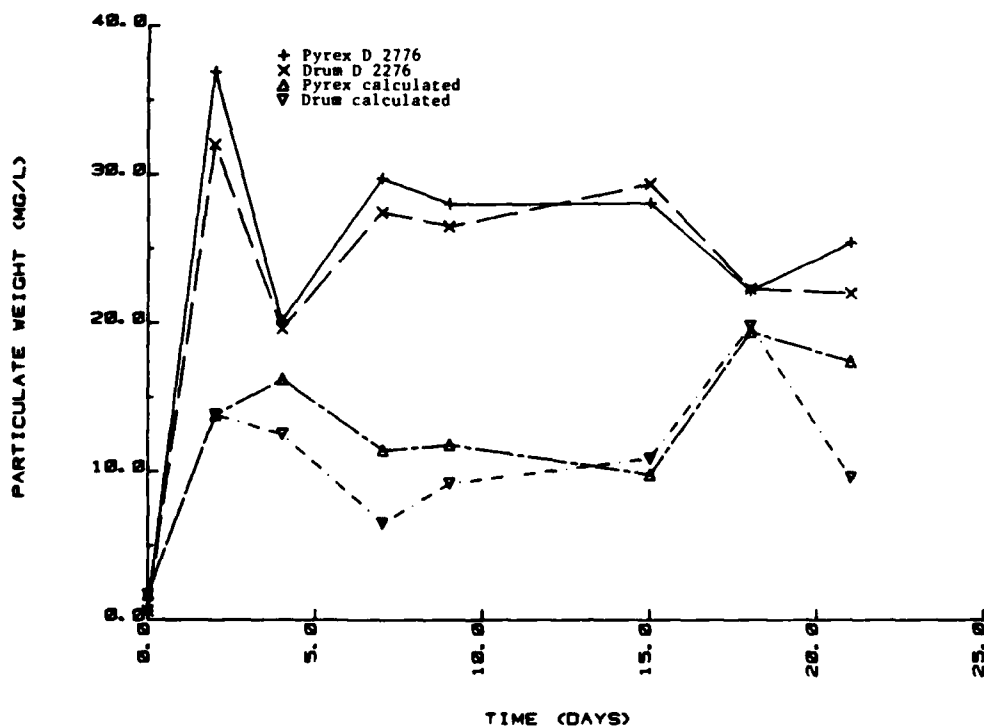


FIGURE 15. COMPARISON OF CALCULATED AND MEASURED PARTICULATE WEIGHT FOR ADDITIVE-FREE CAT 1-H AGED AT 80°C

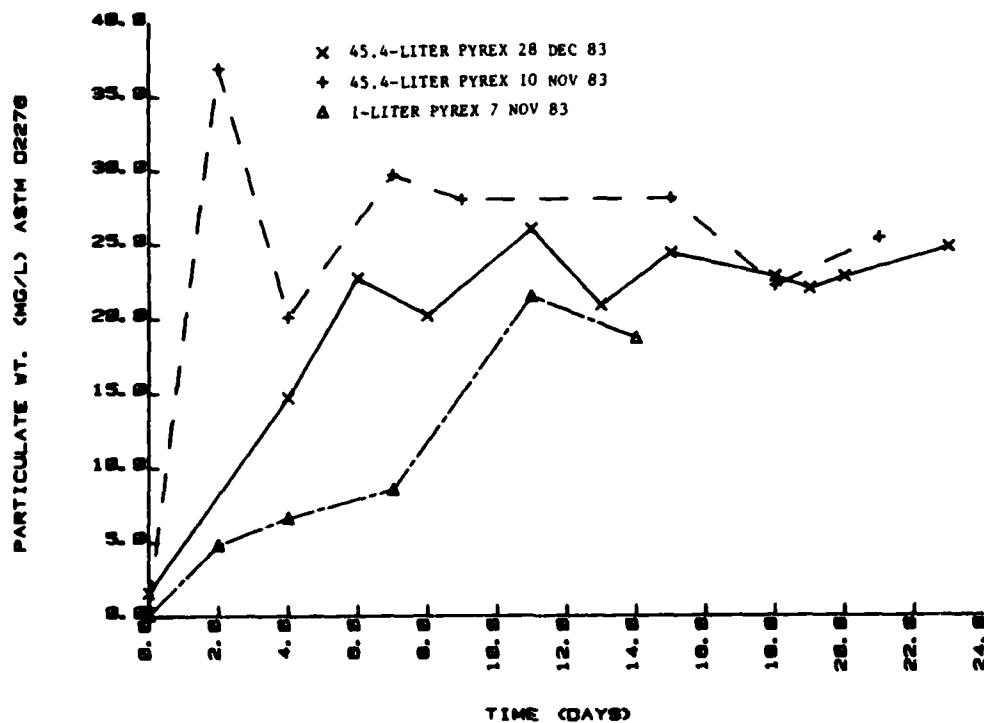


FIGURE 16. MEASURED PARTICULATE WEIGHTS VERSUS TIME FOR ADDITIVE-FREE CAT 1-H AGED AT 80°C IN PYREX® CONTAINERS

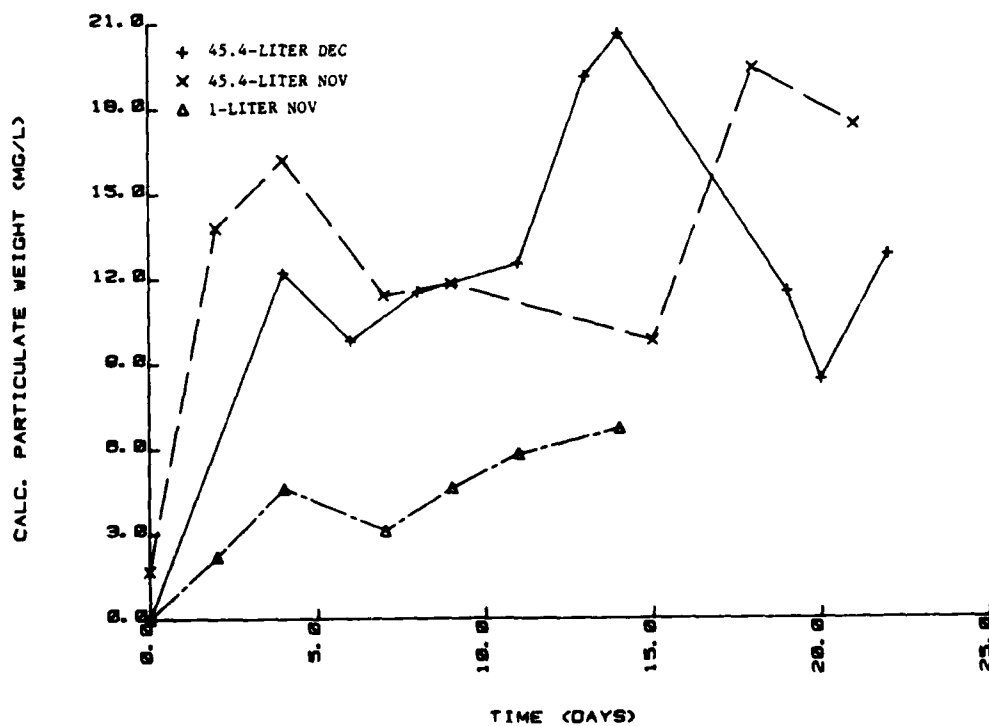


FIGURE 17. CALCULATED PARTICULATE WEIGHT AS A FUNCTION OF TIME FOR ADDITIVE-FREE CAT 1-H AGED AT 80°C IN PYREX® CONTAINERS

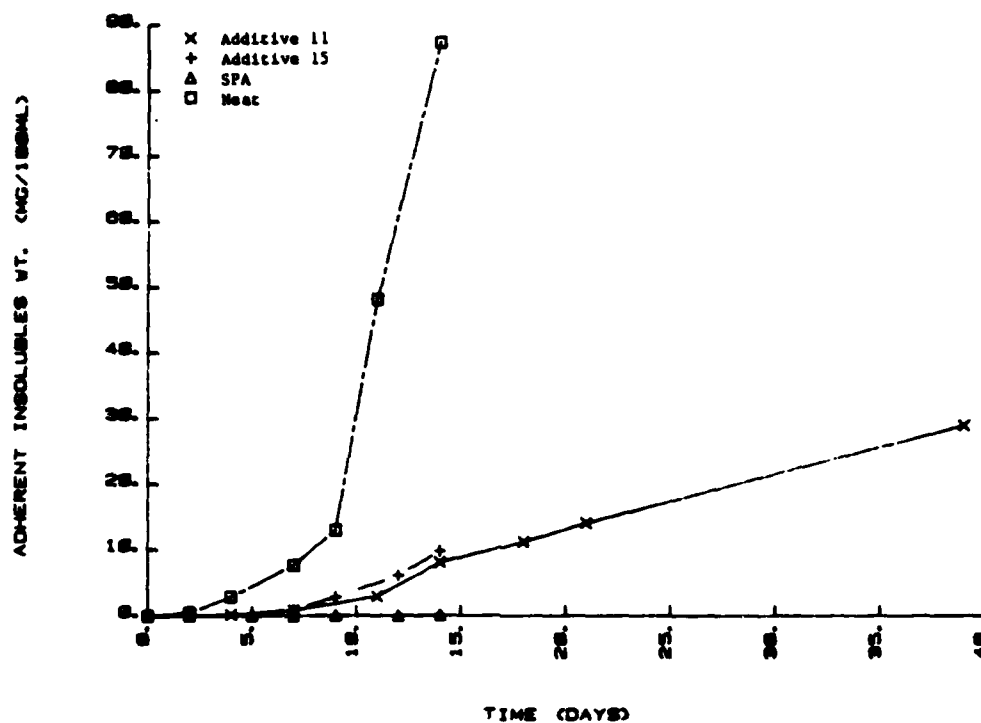


FIGURE 18. COMPARISON OF ADHERENT INSOLUBLES FORMATION FOR CAT 1-H AGED AT 80°C WITH VARIOUS ADDITIVES PRESENT

4. Neat Cat 1-H Fuel at 65°C

The 5-month period (Dec. 1983-May 1984) between tests again allowed a time-dependent measure of repeatability. Except for one point at 7 days in the D 2276 data, all values repeated well. Adherent insolubles weight appeared to be higher by an average factor of about 3 for the older sample. The induction period of 4 days is the same for both experiments. These data are given in Figures 19 and 20. Elemental data are given in Table 4.

5. Cat 1-H With Additive-15 + Additive-B and Single-Package Additive

In each of the tests performed, values obtained for the single-package additive (SPA) were essentially the same as or less than the values obtained for the Additive-15 treated fuel. The tests included filterable particulate weight by ASTM D 2276, calculated particulate weight, adherent insolubles weight, and peroxide number (see also Cat 1-H + Additive-11). The data

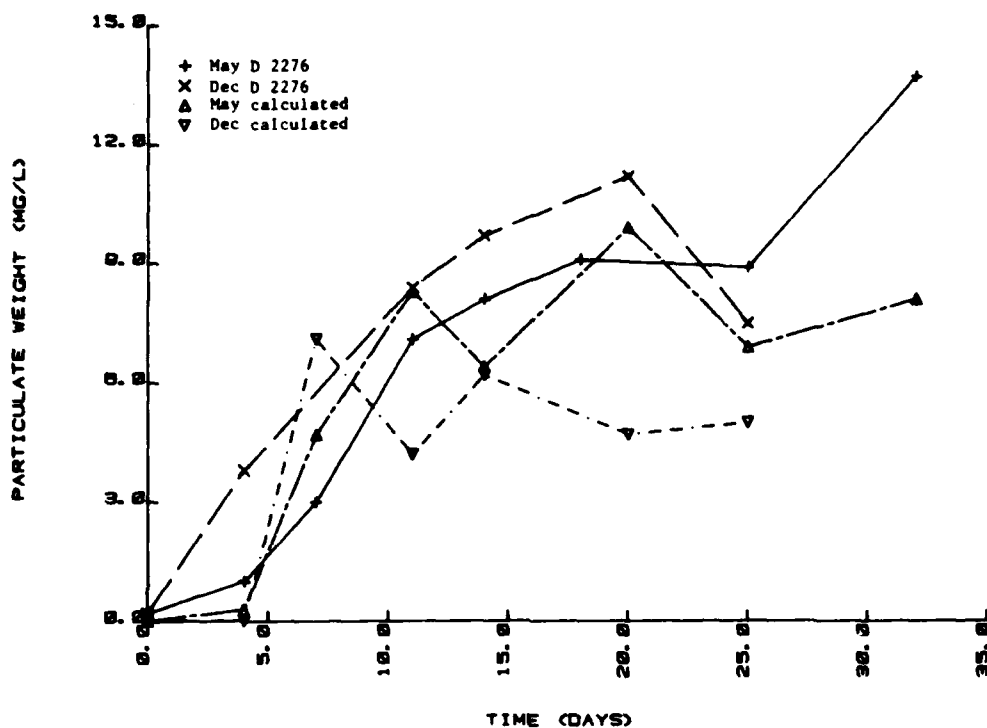


FIGURE 19. COMPARISON OF CALCULATED AND MEASURED PARTICULATE WEIGHT FOR ADDITIVE-FREE CAT 1-H AGED AT 65°C

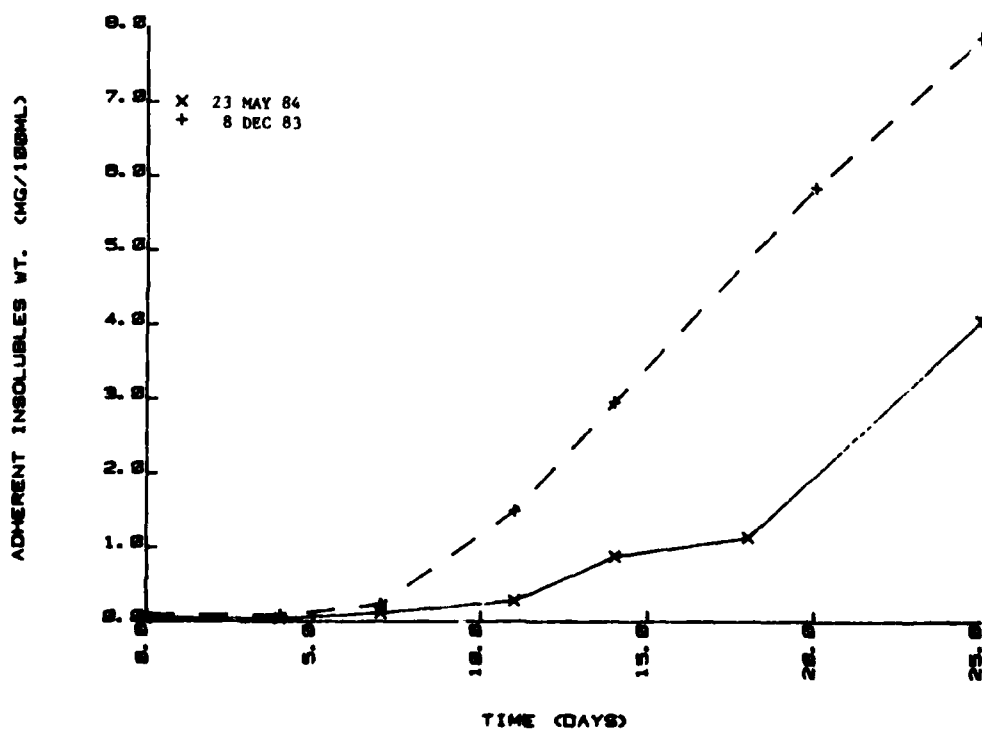


FIGURE 20. ADHERENT INSOLUBLE FORMATION VERSUS TIME FOR ADDITIVE-FREE CAT 1-H AGED AT 65°C

generated are shown in Figures 21 and 22. Insufficient adherent insolubles were formed to allow determination of N or S composition in the SPA-treated fuel. A value for %N in adherent insolubles from the Additive-15/Additive-B test is given in Table 4. A comparison of adherent insolubles formed from Additive-11, Additive-15, SPA-treated, and neat Cat 1-H is shown in Figure 18.

IV. CONCLUSIONS

1. It is possible to model mathematically the ASTM D 2276 gravimetric filterable particulate analysis using particle size and number distribution data.
2. The assumption of a spherical geometry for the particulates may be incorrect because of the consistently greater results for gravimetric data. The difference factor is greater than could be reasonably expected from a density variation (an increase from 2 to 10 times the assumed density would be required for data to be equivalent within a factor of 2).
3. Measurable quantities of fuel-insoluble particulate matter generally form before measurable quantities of adherent fuel insoluble materials, indicating that particulate matter is present as a possible precursor to adherent insolubles.
4. The weight of particulate matter formed in smaller containers appears less than that formed in larger containers, which could indicate an inverse relationship to the contact surface area/volume ratio. Adherent insolubles formation was not monitored in the larger containers as part of this program. A relative increase in adherent insolubles weight in the smaller containers would verify Item 3 above.
5. Use of fuel additives appeared to reduce the quantity of filterable particulate matter. The dispersants employed in the additives may have caused particle size to be maintained below detection limits of the ex-

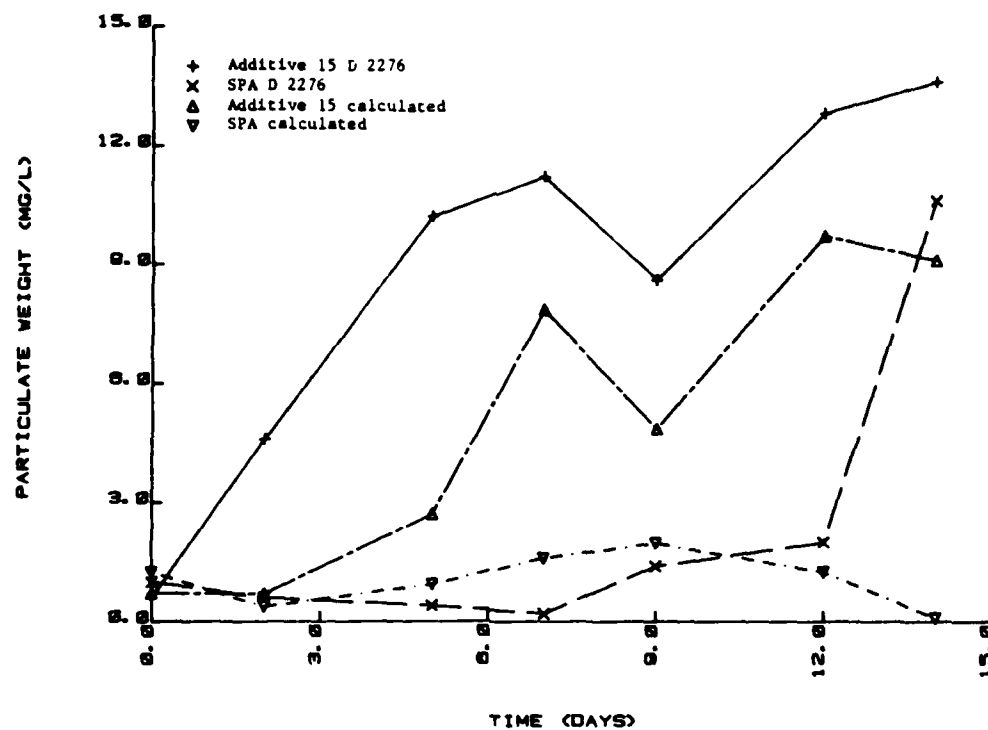


FIGURE 21. COMPARISON OF CALCULATED AND MEASURED PARTICULATE WEIGHT FOR ADDITIVE-TREATED CAT 1-H

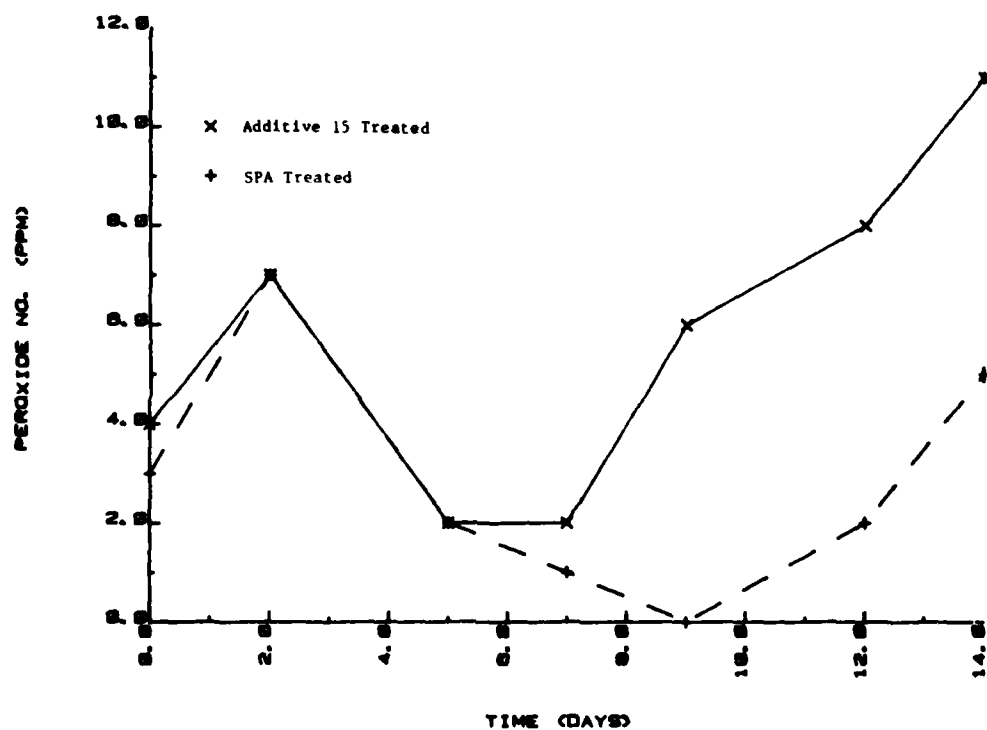


FIGURE 22. PEROXIDE NUMBER VERSUS TIME FOR ADDITIVE-TREATED CAT 1-H AT 80°C

perimental procedure. Conversely, the quantity of adherent insolubles formed appears greater in the additive-treated fuel tests. This relationship may further verify item 3 above.

6. Repeat experiments up to 5 months apart provide similar data from the same fuel. This indicates that ambient drum storage does not materially affect fuel stability even through the winter/spring period. One exception was adherent insoluble formation being reduced by a factor of 3 in the second test.
7. An additive-treated fuel aged at three separate temperatures (15°C intervals) produced adherent insolubles in proportionately higher quantities. The particulate matter formation at the intermediate temperature did not increase in the anticipated amount, possibly due to slow formation rates or detection limits being too high to properly measure the actual quantity of insoluble particulate matter formed.
8. Nitrogen and sulfur-containing chemical species are major factors in fuel-insoluble product formation. The concentration of these species in the adherent insolubles can be many orders of magnitude higher than in the fuel. Nitrogen is more pronounced in this respect than sulfur.
9. Although values for peroxide number reflect changes as the fuel is aged, interpretation of these results is difficult because of their erratic nature.
10. Bromine number, reflecting olefinic bonding, remains constant, indicating a lesser role for double bonds in the overall reaction sequence than expected.
11. Natural sunlight, even through reflection, has a major effect on fuel deterioration studies.

V. RECOMMENDATIONS

Recommendations for continuing investigation into the mechanism of middle distillate fuel determinations and additive inhibitors are:

1. Investigate the role of dissolved oxygen on particulate and adherent gum growth through isotopic labeling ($^{18}\text{O}_2$).
2. Chemically cleave the highly polar/nonvolatile portions of the deterioration products to allow passage through GC columns for identification purposes.
3. Investigate/employ pyrolysis techniques for volatilization/identification of the deterioration products.
4. Place a greater emphasis on the isolation/identification of existent gums and their role in fuel insoluble gum formation. Relate these findings to studies in other laboratories using single-component systems.
5. Continue energy of activation studies and relate findings to fuel and gum compositions.
6. Investigate adherent insoluble formation as a function of surface area/fuel volume ratio to determine the degree to which wall effects influence insoluble product formation.

VI. LIST OF REFERENCES

1. Garner, M.Q. and White, E.W., "The Storage Stability of Navy Distillate Fuel for Ships--Final Report," Report 4198, Department of the Navy, Naval Ship Research and Development Center, 1974.

2. Stavinocha, L.L., Westbrook, S.R., and LePera, M.E., "Army Needs for Diesel Fuel Stability and Cleanliness," Distillate Fuel Stability and Cleanliness, ASTM STP 75, L.L. Stavinocha and C.P. Henry, Eds., ASTM, pp. 103-125, 1981.
3. "Diesel Fuel Stability and Cleanliness Problems in the Field," A Continuous Listing of Fuel Stability Related Problems at Various Military Installations Through 1984 - U.S. Army Belvoir R&D Center, STRBE-VF, Ft. Belvoir, VA.
4. Gruse, W.A. and Stevens, D.R., "The Chemical Technology of Petroleum," McGraw-Hill Book Company, 2nd Edition, 1942.
5. Sauer, R.W., Weed, A.F., and Headington, C.E., "A Mechanism for Organic Sediment Formation in Heating Oils," Petroleum Division Preprints, ACS Meeting, Chicago, IL, September 1958.
6. Frankenfeld, J.W. and Taylor, W.F., "Fundamental Synthetic Fuel Stability Study," Final Report, DOE Contract DE-AC19-79BC10045, March 1982.
7. Nowack, C.J., et al., "Relation Between Fuel Properties and Chemical Composition. IV Stability of Oil Shale Derived Jet Fuel," ACS, Division of Fuel Chemistry Preprints, 25 (3), pp. 40-50, 1980.
8. Worstell, J.H., Daniel, S.R., and Fraunhoff, G., "Deposit Formation in Liquid Fuels. 3. The Effect of Selected Nitrogen Compounds on Diesel Fuel," Fuel, 60, pp. 485-7, 1981.
9. Mayo, F.R. and Buttrill, S.E., Jr., "Chemistry of Fuel Deposits and Sediments and Their Precursors," Final Report to NASA-Lewis Research Center, SRI Project No. PYU 2115, September 1981.
10. Brinkman, D.W., Bowden, J.N., Frankenfeld, J., and Taylor, B., "Synfuel Stability: Degradation Mechanisms and Actual Findings."
11. Nowack, C.J., et al., "Relation Between Fuel Properties and Chemical Composition," ACS Division of Fuel Chemistry Preprints, 25 (3), pp. 40-50, August 1980.
12. Hazlett, R.N. and Hall, J.M., "Studies to Develop a Test to Determine the Potential of a Fuel for Hydroperoxide Formation," CRC Group on Oxidation Stability of Gas Turbine Fuels, 1 May 1981.
13. Hazlett, R.N., et al., "Mechanisms of Syncrude/Synfuel Degradation; Progress Report; NRL Problem #61-1463-00-02, February 1982.

14. Jones, L., et al., "Storage Stability Studies of Fuels Derived From Shale and Petroleum," FUEL, 63 (8), August 1984.
15. Lee, G. H., II, and Stavinocha, L.L., "AFLRL Basic Research on Fuel Storage Stability," Army Research Office, Engines/Fuels Workshop, Contract No. DAAK70-82-C-0001, AD A122843, San Antonio, TX, 6-8 December 1982.
16. Lee, G. H., II, "Status Review of AFLRL Basic Research on Fuel Storage Stability," Army Research Office, Program Review, Contract No. DAAK70-82-C-0001, Ft. Belvoir, VA, 15 May 1984.
17. Lee, G.H., II, and Stavinocha, L.L., "Insoluble Gum Formation in Middle Distillate Fuels," Conference on Long-Term Storage Stabilities of Liquid Fuels," Tel Aviv, Israel, 12 July 1983.
18. Lee, G.H., II, and Stavinocha, L.L., "Middle Distillate Fuel Stability Characteristics--A Review," Interim Report AFLRL No. 168, AD A132156, prepared by U.S. Army Fuels and Lubricants Research Laboratory under Contract No. DAAK70-82-C-0001, January 1983.

DISTRIBUTION LIST

DEPARTMENT OF DEFENSE

DEFENSE DOCUMENTATION CTR
CAMERON STATION 12
ALEXANDRIA VA 22314

DEPT. OF DEFENSE
ATTN: DASD-LM(ET)PE (MR DYCKMAN) 1
WASHINGTON DC 20301

CDR
DEFENSE FUEL SUPPLY CTR
ATTN: DFSC-T (MR MARTIN) 1
CAMERON STATION
ALEXANDRIA VA 22304-6160

DEFENSE ADVANCED RES PROJ
AGENCY
DEFENSE SCIENCES OFC 1
1400 WILSON BLVD
ARLINGTON VA 22209

DEPARTMENT OF THE ARMY

HG, DEPT OF ARMY
ATTN: DALO-TSE (LTC BLISS) 1
DAMA-ARZ-E (DR CHURCH) 1
DAMA-ART (LTC RINEHART) 1
WASHINGTON DC 20310

CDR
U.S. ARMY BELVOIR RESEARCH AND
DEVELOPMENT CENTER
ATTN: STRBE-VF 10
STRBE-WC 2
FORT BELVOIR VA 22060-5606

CDR
US ARMY MATERIEL DEVEL &
READINESS COMMAND
ATTN: AMCLD (DR ODOM) 1
AMCQA-E 1
5001 EISENHOWER AVENUE
ALEXANDRIA VA 22333-0001

CDR
US ARMY TANK-AUTOMOTIVE CMD
ATTN: AMSTA-RG (MR WHELOCK) 1
AMSTA-GBP (MR MCCARTNEY) 1
AMSTA-RC 1
WARREN MI 48397-5000

DIRECTOR
US ARMY MATERIEL SYSTEMS
ANALYSIS ACTIVITY
ATTN: AMXSY-CM (MR NIEMEYER) 1
AMXSY-CR 1
ABERDEEN PROVING GROUND MD 21005

DIRECTOR
APPLIED TECHNOLOGY LAB
U.S. ARMY R&T LAB (AVSCOM)
ATTN: SAVDL-ATL-ATP (MR MORROW) 1
FORT EUSTIS VA 23604

CDR
US ARMY GENERAL MATERIAL &
PETROLEUM ACTIVITY
ATTN: STRGP-F (MR ASHBROOK) 1
STRGP-PE, BLDG 85-3 1
NEW CUMBERLAND PA 17070-5008

CDR
US ARMY RES & STDZN GROUP
(EUROPE)
ATTN: AMXSN-UK-RA (DR OERTEL) 1
BOX 65
FPO NEW YORK 09510

CDR, US ARMY AVIATION R&D CMD
ATTN: AMSAV-EP (MR EDWARDS) 1
4300 GOODFELLOW BLVD
ST LOUIS MO 63120-1798

CDR
US ARMY FORCES COMMAND
ATTN: AFLG-REG 1
AFLG-POP 1
FORT MCPHERSON GA 30330

CDR
US CENTRAL COMMAND
ATTN: CINCCEN/CC J4-L 1
MACDILL AIR FORCE BASE FL 33608

CDR
US ARMY YUMA PROVING GROUND
ATTN: STEYP-MLS-M (MR DOEBBLER) 1
YUMA AZ 85364

PROJ MGR, MOBILE ELECTRIC POWER
ATTN: AMCPM-MEP-TM 1
7500 BACKLICK ROAD
SPRINGFIELD VA 22150

CDR		HQ, EUROPEAN COMMAND	
US ARMY EUROPE & SEVENTH ARMY		ATTN: J4/7-LJPO (LTC LETTERIE)	1
ATTN: AEAGG-FMD	1	VAIHINGEN, GE	
AEAGD-TE	1	APO NY 09128	
APO NY 09403			
CDR		CDR	
THEATER ARMY MATERIAL MGMT		US ARMY GENERAL MATERIAL &	
CENTER (200TH)-DPGM		PETROLEUM ACTIVITY	
DIRECTORATE FOR PETROL MGMT		ATTN: STRGP-PW (MR PRICE)	1
ATTN: AEAGD-MMC-PT-Q	1	BLDG 247, DEFENSE DEPOT TRACY	
APO NY 09052		TRACY CA 95376	
CDR		CDR	
US ARMY RESEARCH OFC		US ARMY FOREIGN SCIENCE & TECH	
ATTN: AMXRO-ZC	1	CENTER	
AMXRO-EG (DR MANN)	1	ATTN: AMXST-MT-1	1
AMXRO-CB	1	AMXST-BA	1
PO BOX 12211		FEDERAL BLDG	
RSCH TRIANGLE PARK NC 27709-2211		CHARLOTTESVILLE VA 22901	
DIR		HQ, US ARMY T&E COMMAND	
US ARMY AVIATION R&T LAB		ATTN: AMSTE-TO-O	1
(AVRADCOM)		ABERDEEN PROVING GROUND MD 21005-5006	
ATTN: SAVDL-AS (MR WILSTEAD)	1	CDR, US ARMY TROOP SUPPORT	
AMES RSCH CTR		COMMAND	
MAIL STP 207-5		ATTN: AMSTR-ME	1
MOFFET FIELD CA 94035		4300 GOODFELLOW BLVD	
		ST LOUIS MO 63120	
CDR		CDR	
TRADOC COMBINED ARMS TEST		CONSTRUCTION ENG RSCH LAB	
ACTIVITY		ATTN: CERL-EM	1
ATTN: ATCT-CA	1	CERL-ZT	1
FORT HOOD TX 76544		CERL-EH	1
CDR		PO BOX 4005	
105TH S & T BATTALION		CHAMPAIGN IL 61820	
5TH INFANTRY DIV (MECH)	1	TRADOC LIAISON OFFICE	
FORT POLK LA 71459		ATTN: ATFE-LO-AV	1
CDR		4300 GOODFELLOW BLVD	
TOBYHANNA ARMY DEPOT		ST LOUIS MO 63120	
ATTN: SDSTO-TP-S	1	HQ	
TOBYHANNA PA 18466		US ARMY TRAINING & DOCTRINE CMD	
CDR		ATTN: ATCD-SL-5 (MAJ JONES)	1
US ARMY DEPOT SYSTEMS CMD		FORT MONROE VA 23651-5000	
ATTN: AMSDS-RM-EFO	1	DIRECTOR	
CHAMBERSBURG PA 17201		US ARMY RSCH & TECH LAB	
CDR		(AVRADCOM)	
US ARMY LEA		PROPULSION LABORATORY	
ATTN: DALO-LEP	1	ATTN: SAVDL-PL-D (MR ACURIO)	1
NEW CUMBERLAND ARMY DEPOT		21000 BROOKPARK ROAD	
NEW CUMBERLAND PA 17070		CLEVELAND OH 44135	

CDR
US ARMY NATICK RES & DEV LAB
ATTN: STRNA-YE (DR KAPLAN) 1
STRNA-U 1
NATICK MA 01760

CDR
US ARMY QUARTERMASTER SCHOOL
ATTN: ATSM-CD 1
ATSM-TD 1
ATSM-PFS 1
FORT LEE VA 23801

CDR
COMBINED ARMS COMBAT
DEVELOPMENT ACTIVITY
ATTN: ATZL-CAT-E 1
ATZL-CAT-A 1
FORT LEAVENWORTH KA 66027-5300

CDR
US ARMY LOGISTICS CTR
ATTN: ATCL-MS (MR A MARSHALL) 1
ATCL-C 1
FORT LEE VA 23801-6000

PROJECT MANAGER
PETROLEUM & WATER SYSTEMS
ATTN: AMCPM-PWS 1
4300 GOODFELLO BLVD
ST LOUIS MO 63120

CDR
US ARMY AVIATION CTR & FT RUCKER
ATTN: ATZQ-DI 1
FORT RUCKER AL 36362

CDR
6TH MATERIEL MANAGEMENT CENTER 1
19TH SUPPORT BRIGADE
APO SAN FRANCISCO 96212

CDR
US ARMY SAFETY CENTER
ATTN: PESQ-SSD (MR BUCHAN) 1
FORT RUCKER AL 36362

DEPARTMENT OF THE NAVY

CDR
NAVAL AIR PROPULSION CENTER
ATTN: PE-33 (MR D'ORAZIO) 1
PO BOX 7176
TRENTON NJ 06828

CDR
NAVAL SEA SYSTEMS CMD
ATTN: CODE 05M4 (MR R LAYNE) 1
WASHINGTON DC 20362

CDR
DAVID TAYLOR NAVAL SHIP R&D CTR
ATTN: CODE 2830 (MR BOSMAJIAN) 1
CODE 2759 (MR STRUCKO) 1
CODE 2831 1
ANNAPOLIS MD 21402

CDR
NAVAL SHIP ENGINEERING CENTER
ATTN: CODE 6764 1
PHILADELPHIA PA 19112

JOINT OIL ANALYSIS PROGRAM -
TECHNICAL SUPPORT CTR 1
BLDG 780
NAVAL AIR STATION
PENSACOLA FL 32508

DEPARTMENT OF THE NAVY
HQ, US MARINE CORPS
ATTN: LPP (MAJ WALLER) 1
WASHINGTON DC 20380

CDR
NAVAL AIR SYSTEMS CMD
ATTN: CODE 53645 (MR MEARNES) 1
WASHINGTON DC 20361

CDR
NAVAL RESEARCH LABORATORY
ATTN: CODE 6170 1
CODE 6180 1
CODE 6110 (DR HARVEY) 1
WASHINGTON DC 20375

COMMANDING GENERAL
US MARINE CORPS DEVELOPMENT
& EDUCATION COMMAND
ATTN: D074 (LTC WOODHEAD) 1
QUANTICO VA 22134

OFFICE OF CHIEF OF NAVAL
RESEARCH
ATTN: ONT-07E (MR ZIEM) 1
ARLINGTON, VA 22217

CHIEF OF NAVAL OPERATIONS
ATTN: OP 413 1
WASHINGTON DC 20350

CDR
NAVY PETROLEUM OFC
ATTN: CODE 43
CAMERON STATION
ALEXANDRIA VA 22314

1

US DEPARTMENT OF ENERGY
CE-1312
ATTN: MR ECKLUND
FORRESTAL BLDG.
1000 INDEPENDENCE AVENUE, SW
WASHINGTON DC 20585

1

DEPARTMENT OF THE AIR FORCE

HQ, USAF
ATTN: LEYSF (COL CUSTER)
WASHINGTON DC 20330

1

ENVIRONMENTAL PROTECTION AGENCY
AIR POLLUTION CONTROL
2565 PLYMOUTH ROAD
ANN ARBOR MI 48105

1

HQ AIR FORCE SYSTEMS CMD
ATTN: AFSC/DLF (MAJ VONEDA)
ANDREWS AFB MD 20334

1

CDR
US AIR FORCE WRIGHT AERONAUTICAL
LAB
ATTN: AFWAL/POSF (MR CHURCHILL)
WRIGHT-PATTERSON AFB OH 45433

1

CDR
SAN ANTONIO AIR LOGISTICS CTR
ATTN: SAALC/SFT (MR MAKRIS)
SAALC/MMPRR
KELLY AIR FORCE BASE TX 78241

1

1

CDR
HQ 3RD USAF
ATTN: LGSF (MR PINZOLA)
APO NEW YORK 09127

CDR
DET 29
ATTN: SA-ALC/SFM
CAMERON STATION
ALEXANDRIA VA 22314

1

OTHER GOVERNMENT AGENCIES

DEPARTMENT OF TRANSPORTATION
FEDERAL AVIATION ADMINISTRATION
ATTN: AWS-110
800 INDEPENDENCE AVENUE, SW
WASHINGTON DC 20590

1

END

FILMED

12-85

DTIC